AFOSR-TR-97

0284

REPORT DOCUMENTATION PAGE

1. AGENCY USE ONLY (Leave blank)	2. REPORT DATE November 18, 1996	3. REPORT TYPE AND DATES COVERED Final (8/1/95-12/31/96)

4. TITLE AND SUBTITLE

Durability and Life Asessment for Affordable Composites Manufacturing of Polymer-Matrix Composites

5. FUNDING NUMBERS

F49620-95-1-0486

6. AUTHOR(S)

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8. PERFORMING ORGANIZATION REPORT NUMBER

3-3-21-3144-48

9. SPONSORING/MONITORING AGENCY NAMES(S) AND ADDRESS(ES)

AFOSR/PKA
110 Duncan Avenue Room B115
Bolling AFB, DC 20332-8080

10. SPONSORING/MONITORING AGENCY REPORT NUMBER

Project-Task 3484/US

11. SUPPLEMENTARY NOTES

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12a. DISTRIBUTION/AVAILABILITY STATEMENT

Approved for public release; distribution unlimited.

12B. DISTRIBUTION CODE

13. ABSTRACT (Maximum 200 words)

The capability to characterize and test advanced composites in simulated operational environments—including elevated temperatures, thermal cycling, moisture, oxidation, solvents, etc.—is essential to the future use of these material systems in various civilian and military applications. This program sought funding to establish core facilities for durability assessment of advanced polymer-matrix composites in such applications as bridge rehabilitation, high-speed civil aircraft, engine components for propulsion, and armored ground vehicles. The cross-cutting issue in these programs is the durability of composite materials. The equipment purchased is being used to promote (1) significant advances in the fundamental understanding of degradation mechanisms and (2) concomitant improvements in design and processing aimed at increasing the durability of composite components for these applications.Researchers are currently using the equipment to measure changes in local elasticity over a sample surface. The results obtained thus far show promising correlation between the generated force curve data and the sample moduli. The work has successfully extended the capabilities of the AFM as a probe of nanomechanical properties and property variations important to the performance of composite materials and adhesive. It has also resulted in development of the first experimental technique to directly examine interphase regions in multiphase systems.

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DTIC QUALITY INSPECTED 3

15. NUMBER OF PAGES

4

16. PRICE CODE

17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT
UNCLASSIFIED	UNCLASSIFIED	UNCLASSIFIED	UL

NSN 7540-01-280-5500

Standard Form 298 (Rev. 2-89)
Prescribed by ANSI Std. 239-18
298-102

1995–96 AFOSR
Defense University Research Instrumentation Program
(DURIP)

Durability and Life Assessment for Affordable Manufacturing of Polymer-Matrix Composites

Contract/Award: F49620-95-1-0486
August 1, 1995–December 31, 1996

UNIVERSITY OF DELAWARE
CENTER FOR COMPOSITE MATERIALS
INTERNATIONALLY RECOGNIZED EXCELLENCE

Final Report

November 1996

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Form Approved
OMB No. 0704-0188

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Background/Motivation

The capability to characterize and test advanced composites in simulated operational environments—including elevated temperatures, thermal cycling, moisture, oxidation, solvents, etc.—is essential to the future use of these material systems in various civilian and military applications. This program sought funding to establish core facilities for durability assessment of advanced polymer-matrix composites in such applications as bridge rehabilitation, high-speed civil aircraft, engine components for propulsion, and armored ground vehicles. The cross-cutting issue in these programs is the durability of composite materials. The equipment purchased is being used to promote (1) significant advances in the fundamental understanding of degradation mechanisms and (2) concomitant improvements in design and processing aimed at increasing the durability of composite components for these applications.

Elucidation of degradation mechanisms requires interrogation of composites at both the microscopic and macroscopic levels, with a particular emphasis on issues related to the fiber/matrix interface. The interface—or more correctly, the *interphase*, as it is actually a region—has been proposed to nucleate microcracks in composites, which evolve into macroscopic failure. The interphase is also a conduit for the ingress of moisture, which, during repeated thermal cyclings, causes expansion and contraction of the composite, leading to failure. In short, what happens at the microscopic level permits aging at an accelerated pace. Thus, the instrumentation purchased with this grant is enabling these mechanisms to be investigated both in isolation and in combination to provide an overall profile of composite durability.

Equipment Purchased	
B142786 Dimension 3000 Scanning Probe Microscope	43,630.00
B227710 3 AT-GPIB/TNT-488 Interface Card for Windows 95 w/2 Meter Cable, 1 Labview for Windows 95 Base Package, Shipping	$2,215.50
B238250 Tenney Environmental Chamber	$17,230.09
B226190 3-Dimension XPS P133S Computer System	$7,662.00
Upgrade to Testing Lab, including Instron Model 4484 floor-mounted materials testing system	$154,257.00
Total:	$224,995.59

Research Issues

Long-term environmental concerns include the effects of heat aging, degradation from natural elements (i.e., rain/humidity, salt, UV radiation), effects of service environmental fluids (e.g., fuels, oils, deicers, paint strippers). It has been shown that humidity effects can severely reduce mechanical properties, and they typically have a synergistic effect with temperature (i.e., there is a greater effect at elevated temperatures). Exposure to moisture can result in transverse matrix cracking in cross-ply and quasi-isotropic laminates. The glass transition temperature (T_g), fracture toughness (G_{IC}), and bearing strength are all significantly affected by moisture; upon drying, T_g and bearing strength are regained, but G_{IC} is not, indicating permanent damage.

On the microscopic level, the behavior of a formed part is affected by the formation of interphase zones that can lead to variations in local material properties such as modulus and glass transition temperature. Such compositional and material property gradients affect microcracking and interfacial strength, which influence macroscopic characteristics such as strength and fracture toughness.

The unifying concept is that complex material systems can be treated as a hierarchy of structures associated with decreasing scales. Each level of structure imparts certain behavior to the next succeeding higher level of organization, and each exhibits differing response sensitivities to the environments encountered under service conditions. This approach offers the potential for translating service condition requirements to the molecular level.

Semi-empirical analyses conducted at the gross structural level of composites have been relatively successful in predicting and describing effective thermomechanical behavior in terms of the properties, concentration, and geometry of the components. These properties are manifestations of bulk average behavior, which is insensitive to localized perturbations. Similar treatments fail to adequately predict and describe ultimate properties, fracture, fatigue performance, impact strength, or environmental effects. Properties belonging to this important group are sensitive to the "weak links" generated by small-scale variations in structure and properties. Progress in understanding this vital class of properties requires research directed at a sublevel of organization tentatively characterized as a microsystem. It is at this structural level that the interphase becomes important.

A focus on this reduced scale can take into account inhomogeneities in the internal structure of the reinforcing agents and the matrix, as well as the possibility of a perturbed interphase region near the surface of the reinforcing agent. It is generally accepted that the nature of the interphase region can be dependent upon the chemical composition of the fiber and matrix as well as that of the coupling agents and additives. The behavior of the interphase region is expected to be sensitive to concentration and thermal gradients encountered during processing.

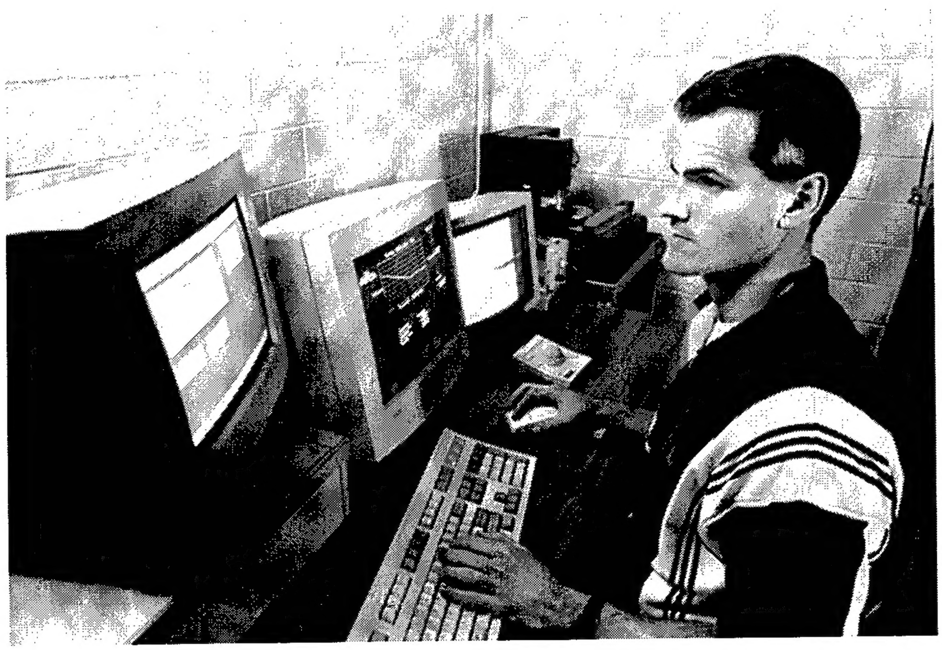
The response of candidate materials for these applications under operating conditions needs to be characterized over the expected life cycle, which in all cases is long. The inordinate time and expense involved in such testing necessitates the development of accelerated test methodologies to address the durability issues. Verification of such methodologies once they have been developed will necessitate facilities enabling temperature and mechanical fatigue to be varied; additional variables to be investigated include the presence of moisture and chemical solvents. In other words, there is an urgent need for the capability to look at the synergistic superposition of service load cases within a single testing environment.

Current Research Using Purchased Equipment

The atomic force microscope (AFM) is being used to investigate environmental effects—including elevated-temperature as well as high humidity—on the properties and behavior of polymer-matrix composites. It is known that these environments reduce composite properties, and it is assumed that this property reduction is due to degradation of the fiber/matrix interphase. However, while theoretical work has been done, models have been developed, and indirect methods have supported this assumption, it has not been verified experimentally; the AFM is a powerful new tool in the effort to do so.

Researchers are currently using the equipment to measure changes in local elasticity over a sample surface. Appropriate analysis of AFM force curves enables a great deal of information to be obtained regarding the mechanical, chemical, and adhesive properties of the surface. To date, very little effort has been dedicated to examining the contact portion of the force curve. This region contains valuable information about the nanoscale mechanical response of the sample. A technique is being developed that relates the elastic modulus of the sample to the sample response measured using AFM force curves.

The results obtained thus far show promising correlation between the generated force curve data and the sample moduli. The work has successfully extended the capabilities of the AFM as a probe of nanomechanical properties and property variations important to the performance of composite materials and adhesive. It has also resulted in development of the first experimental technique to directly examine interphase regions in multiphase systems.



Graduate student Mark VanLandingham analyzes force curve data generated by the atomic force microscope purchased by the University of Delaware Center for Composite Materials with AFOSR DURIP funds.

Related Publications

"Characterization and Mechanical Properties of the Epoxy/Polysulfone Interphase," with K. M. Immordino, S. H. McKnight, and M. R. VanLandingham, to be submitted, 1996.

"Nanoscale Indentation of Polymer Systems Using the Atomic Force Microscope," with M. R. VanLandingham, S. H. McKnight, G. R. Palmese, X. Huang, and R. F. Eduljee, submitted to *The Journal of Adhesion*, October 1996.

"Characterization of the Elastic Properties of the Fiber-Matrix Interphase Using the Atomic Force Microscope," with R. F. Eduljee and M. R. Van Landingham, submitted to *The Journal of Adhesion*, 1996.

"Relating Elastic Modulus to Indentation Response Using Atomic Force Microscopy," with M. R. Van Landingham, S. H. McKnight, G. R. Palmese, R. F. Eduljee and R. L. McCullough, *Proceedings of the American Society for Composites 11th Technical Conference on Composite Materials*, Technomic Publishing Company, Lancaster, PA, 1996.

"Moisture Effects on the Material Behavior in Graphite/Polyimide Composites," with L. J. Burcham, M. R. Van Landingham, and R. F. Eduljee, *Polymer Composites*, Vol. 17, No. 5, pp. 682–690, October 1996.

"The Effect of Hygrothermal Cycling on the Microcracking Behavior of Fabric Laminates," with R. F. Eduljee and M. R. VanLandingham, *Proceedings of the 28th International SAMPE Technical Conference*, Seattle, WA, November 4–7, 1996.